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The Increasing of Water Transport Resources under the Influence of a Thermal Field by the Usage of Modified Epoxy Nanomaterials

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The work represents the results of experimental research on the influence of the nanofiller's nature on the thermal physical properties of composite materials. Polymer composite materials based on epoxy diane oligomer ED-20 were modified by 4-aminobenzoic acid. Martens yield temperature was researched and the glass transition temperature of the developed materials was determined. For the establishment of the thermal field's effect on the structural processes of composite materials, the thermal coefficient of linear expansion of the materials was researched. Due to the necessity of studying the influence of temperature on physical-chemical processes and transformations in polymer composites, modern thermal analysis methods were used – differential thermal and thermal gravimetric analysis. Based on the analysis of experimental data, the optimal content of 4-aminobenzoic acid $q = 0.10$ wt.% was determined per 100 wt.% of the ED-20 oligomer, which leads to the improvement of thermal physical properties of epoxy composite materials.

Keywords: Epoxy composite, Epoxy binder, Nanofillers, Nanoscale additives, Heat tesistance, Thermal coefficient of linear expansion, Thermal resistance.

1. INTRODUCTION

In the ever-advancing landscape of material science, the integration of nanomaterials has emerged as a transformative force, especially in industries where competition thrives, such as shipbuilding, aircraft, and machine building [1, 2]. Nanomaterials, with their unique properties at the nanoscale, have become the focal point of research aimed at enhancing the performance of traditional materials. In this context, epoxy oligomers and their derivative composite materials (CM) have garnered significant attention [3, 4]. These materials, known for their commendable resistance to chemical and thermal processes, as well as their improved stiffness and strength compared to conventional alternatives [5, 6], present a promising foundation for further enhancement.

As we delve into the intricate realm of material development, the synergy between traditional modifiers and nanomaterials emerges as a compelling avenue [1, 7]. Nanoscale additives, such as nanoparticles and nanotubes, are now being explored alongside conventional modifiers to augment the thermal physical properties of epoxy composite materials [8, 9]. This dual approach seeks not only to preserve the advantageous traits of traditional modifiers but also to harness the unique characteristics of nanomaterials for unprecedented advancements in structural and thermal performance [10, 11].

This pursuit becomes increasingly imperative in industries where vehicles, particularly those in river and sea transport, endure prolonged exposure to aggressive environmental factors [12]. Developing innovative materials and protective coatings capable of withstanding such challenges is essential for enhancing the durability and operational characteristics of water transport equipment. Established studies [13, 14] emphasize the efficacy of physically modifying epoxy diane oligomer, and the integration of nanomaterials into this paradigm further broadens the spectrum of possibilities for improvement [15].

Against this backdrop, the deliberate addition of a modifier into the polymer, specifically 4-aminobenzoic acid ($C_7H_7NO_2$), renowned for its active interphase interaction, is contemplated as a strategic move to elevate the thermal physical properties of CM based on an epoxy binder [16, 17]. This interdisciplinary approach, encompassing both nanomaterials and traditional modifiers, holds the promise of ushering in a new era of heightened performance and resilience in epoxy composite materials [18].

The objective of the work is the development of new modified epoxy materials to increase the resource of water transport during its operation under the influence of a thermal field.

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2. MATERIALS AND METHODS

During the formation of CM, the ED-20 brand epoxy diane oligomer (ISO 18280:2010) was selected as the main component. ED-20 binder is characterized by high adhesive and cohesive strength, slight shrinkage and manufacturability when applied to the surface of a complex profile [19].

4-aminobenzoic acid was used as a modifier. The modifier was added into the binder at a content of 0.10 to 2.00 wt.% per 100 wt.% of epoxy oligomer ED-20 (here and further in the text, wt.% are given per 100 wt.% of epoxy oligomer ED-20). The formula of 4-aminobenzoic acid is: $C_7H_7NO_2$ [20].

Polyethylene polyamine PEPA hardener is used for cross-linking of epoxy compositions (TU 6-05-241-202-78), which allows forming materials at room temperatures. PEPA is a low-molecular substance that consists of the following linked units: [-CH_2-CH_2-NH-]n. The hardener was added into the composition at a content of 10 wt.% per 100 wt.% of epoxy oligomer ED-20 [6]. The characteristics of epoxy diane oligomer, modifier and hardener are presented in Table 1.

Table 1 – Characteristics of the components of the epoxy binder, hardener and modifier

Characteristics	Epoxy oligomer ED-20	Modifier 4-aminobenzoic acid (C7H7NO2)	Hardener PEPA
Molecular mass	390…430	137.14	230…250
The content of epoxy groups, %	20.0…22.5	–	–
The content of hydroxyl groups, %	1.25	–	–
Average functionality by epoxy groups, fn	2.0	–	–
The content of carbon, %	–	61.31	–
The content of hydrogen, %	–	5.14	–
The content of nitrogen, %	–	10.21	19.5…22.0
The content of oxygen, %	–	23.33	–
Viscosity, η, Pa·s	13…20	–	0.9
Density, ρ, g/cm^3	1.16	1.37	1.05

The epoxy CM was formed by to the following technology: dosage of components; heating the epoxy resin to a temperature of $T = 353 \pm 2$ K and keeping it at mentioned temperature during time of $\tau = 20 \pm 0.1$ min; hydrodynamic mixing of the oligomer and the modifier until obtaining of homogeneous mixture during the time $\tau = 2 \pm 0.1$ min; ultrasonic treatment (UST) of the composition during time $\tau = 1.5 \pm 0.1$ min; cooling the composition to room temperature during time $\tau = 60 \pm 5$ min; introduction of hardener and mixing of the composition during the time $\tau = 5 \pm 0.1$ min. CM was hardened in accordance with the experimentally established mode: the formation of specimens and their keeping at a period of time $\tau = 12.0 \pm 0.1$ h at temperature $T = 298 \pm 2$ K; heating with a rate of $v = 3$ K/min to the cross-linking temperature $T = 413 \pm 2$ K; keeping the

specimens at mentioned temperature during time of $\tau = 2.0 \pm 0.05$ h; slow cooling to temperature $T = 298 \pm 2$ K. Due to necessity to stabilize the structural processes inside the matrix, the specimens were kept during $\tau = 24$ h in air atmosphere with a temperature of $T = 298 \pm 2$ K, followed by the experimental tests.

The thermal physical properties of CM were researched, especially: Martens Yield temperature, thermal coefficient of linear expansion, heat resistance (DTA and TGA analysis).

Martens Yield temperature of CM was determined in accordance with ISO 75-2:2013. The research methodology consists in determining the temperature at which the test specimen was heated at a rate of $v = 3$ K/min under the action of a constant bending load $F = 5 \pm 0.5$ MPa, as a result of which it is deformed by a given value ($h = 6$ mm).

The thermal coefficient of linear expansion (TCLE) of materials was calculated by the influence curve of relative deformation on temperature, approximating this dependence with an exponential function. The relative deformation was determined by the changing of the specimen's length with an increasing of the temperature under stationary conditions (ISO 11359-2:2021). Specimen's sizes for research: $65 \times 7 \times 7$ mm, the non-parallelism of the polished ends is no more than 0.02 mm. Before the researching, the length of the specimen was measured with accuracy ± 0.01 mm. The rate of temperature rise was $v = 2$ K/min. The deviation of the values during researching of indicators of thermal physical properties of CM (Martens Yield temperature, TCLE) was 4...6 % from the nominal value.

Due to necessity to research the influence of modifier's amount on thermal transformations in composites, the method of thermal gravimetrical (TGA) and differential thermal (DTA) analysis was selected, with usage of the Thermoscan-2 derivatograph. The research was conducted in the temperature range $\Delta T = 298...873$ K, using quartz crucibles for specimens with a volume of $V = 0.5$ cm3. During the researching, the rate of temperature rising was $v = 10$ K/min, while Al_2O_3 ($m = 0.5$ g) was used as a reference substance, the weight of the studied specimen was – $m = 0.3$ g. The temperature determination error was $\Delta T = \pm 1$ K. The accuracy of determining thermal effects is 3 J/g. The accuracy of determining the change in specimen mass – $\Delta m = 0.02$ g.

3. DISCUSSION OF RESEARCH RESULTS

The heat resistance of the modified UST epoxy matrix, was established preliminary and its value is $T = 341$ K. Then, the heat resistance of composites with different contents of the modifier was determined. It should be noted that the content of the $C_7H_7NO_2$ modifier in the studied specimens was changed from $q = 0.10$ wt.% to $q = 2.00$ wt.% per 100 wt.% of epoxy oligomer ED-20.

Experimental studies of the influence of the modifier indicate a significant increase in the heat resistance of CM depending on the content of $C_7H_7NO_2$ (Table 2, Fig. 1). In particular, the content of 4-aminobenzoic acid in amount of $q = 0.10$ wt.%, the value of Martens Yield temperature is $T = 360$ K (the maximum on the influence curve of heat resistance on the content of the modifier), which indicates an increasing in the degree of chemical crosslinking in the epoxy polymer. Increasing the content of the modifier to

q = 0.25 wt.% leads to decreasing in Martens Yield temperature of CM by ΔT = 5 K. It should be noted, that for the content of $C_7H_7NO_2$ in the amount q = 1.00 wt.% and q = 1.50 wt.% recorded a minimal decrease in the indicators of the CM's researched properties (up to T = 353 K). Further increasing in the amount of additive until q = 2.00 wt.% arranges forming of CM with a decreasing Martens Yield temperature to T = 350 K. This indicates a decreasing in the number of reactive functional groups, involved in the polymerization of materials.

One of the most important operational and physical characteristics of CM is the glass transition temperature Tg. The glass transition temperature is the temperature at which a polymer state's alteration from a highly elastic or viscous to the glassy under the influence of a thermal field.

Table 2 – The results of the research of thermal physical properties of CM

Characteristic	The content of 4-aminobenzoic acid ($C_7H_7NO_2$), q, wt.%						
	Matrix	0.10	0.25	0.50	1.00	1.50	2.00
Martens Yield temperature, T, K	341	360	355	353	353	353	350
Glass transition temperature, T_g, K	327	326	327	328	317	295	303
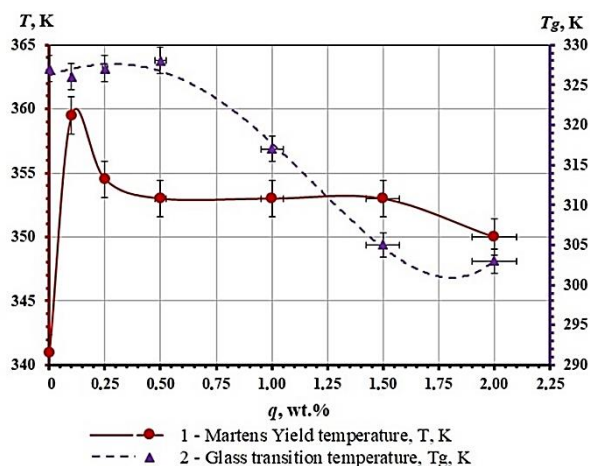
Fig. 1 – Dependence of thermal physical properties of CM on concentrations of the 4-aminobenzoic acid modifier ($C_7H_7NO_2$)

It was established by experiment that the glass transition temperature Tg of the modified UST epoxy matrix is T_g = 327 K. The glass transition temperature of CM, depending on the content of the 4-aminobenzoic acid modifier, is T_g = 295...328 K (Table 2, Fig. 1). At the same time, the value of the studied characteristics of CM according to the content of the modifier q = 0.10 wt.%, q = 0.25 wt.%, q = 0.50 wt.% and q = 1.00 wt.% show almost the same indicators in comparison with the epoxy matrix (T_g = 327 K). It was considered, that the increasing in the glass transition temperature of the epoxy oligomer occurs as a result of an increasing in the density of the chemical network in the modified polymer, as well as due to higher heat resistance indicators of the $C_7H_7NO_2$

modifier compared to the CM matrix. Further increasing in the content of the modifier in the CM, a decreasing in the indicators of thermal physical characteristics was observed. In particular, at the content of the additive q = 1.50 wt.% the glass transition temperature is T_g = 295 K, and at the concentration q = 2.00 wt.% – T_g = 303 K.

In addition, the important technological characteristic is the shrinkage of CM. Shrinkage values of the developed materials were observed within range: δ = 0.11...0.16 % (Table 3, Fig. 2). It does not exceed the value of the pre-modified epoxy matrix (by UST) δ = 0.32 %. The reduction of the materials' shrinkage after the modifier's adding is caused by the physical and chemical interaction of the polymer composition's components during crosslinking. This as a result the thermal stability's increasing and the cracks' probability occurring is decreasing under the action of a thermal field on the CM.

Table 3 – The results of the shrinkages' research

Characteristic	The content of 4-aminobenzoic acid ($C_7H_7NO_2$), q, wt.%						
	Matrix	0.10	0.25	0.50	1.00	1.50	2.00
Shrinkage, δ, %	0.32	0.15	0.16	0.16	0.15	0.11	0.11
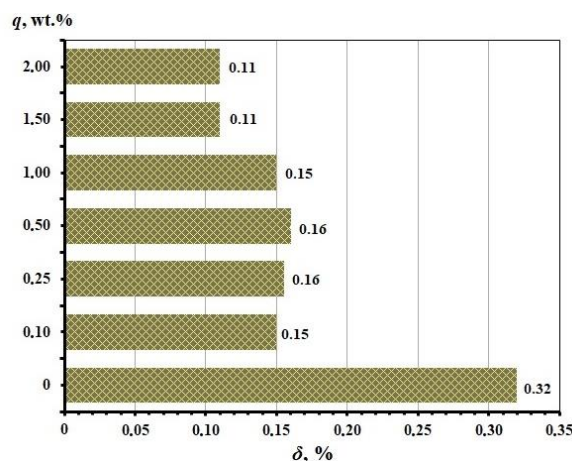
Fig. 2 – The dependence of CM's shrinkage (δ) on concentration of modifier of 4-aminobenzoic acid ($C_7H_7NO_2$)

At the next stage, a research of the thermal coefficient of linear expansion (TCLE) of CM was performed. From a practical point of view, mentioned research is important, as the value of TCLE characterizes the effect of temperature on the expansion of CM, which, in turn, can indicate the reliability of the modified material when the temperature increases.
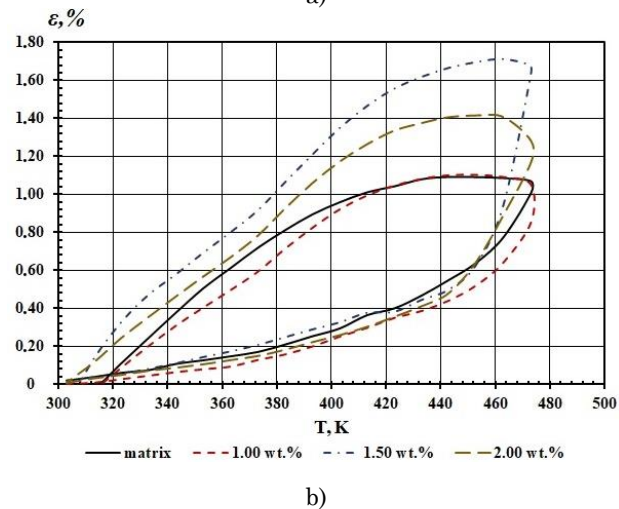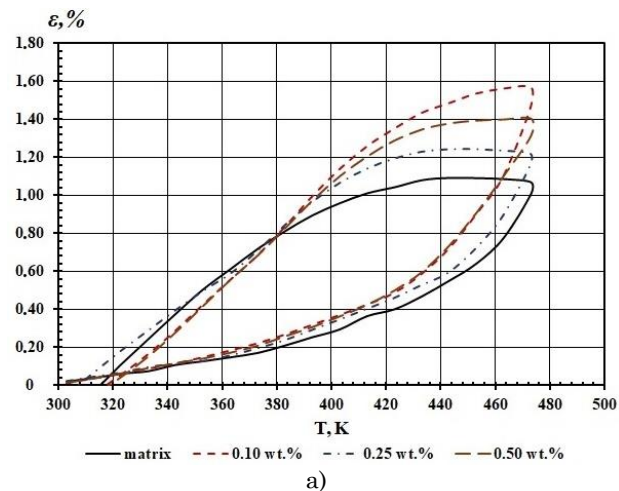
The experimentally obtained data show, that an increasing of the temperature at different ranges of the experiment, an increase in the TCLE of the modified CM was observed (Table 4, Fig. 3). It was established that the values of TCLE of the CM at different concentrations of the modifier do not exceed the values of TCLE of the epoxy matrix. The results of the study of polymeric CM's with the content of a modifier in the amount of q = 1.00 wt.% are especially notable, same as q = 2.00 wt.%, which may indicate the physical and chemical interaction between the components of the binder during the structuring of the matrix.

An equally important property of CM's is their thermal stability – heat resistance. The thermal stability of polymer CM's is the temperature limit up to which the composite retains its initial chemical structure under the influence of a thermal field.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) methods were used to study the heat resistance of CM under conditions of elevated temperatures ($\Delta T = 298\ldots873$ K), while the rate of temperature increase of the epoxy matrix and modified materials was $v = 10$ K/min.

Table 4 – Thermal coefficient of linear expansion (TCLE) of CM at different test temperature ranges

The content of modifier $C_7H_7NO_2$, q, wt.%	Thermal coefficient of linear expansion, $\alpha \times 10^{-5}$, K^{-1}			
	Temperature ranges of experiment, ΔT, K			
	303…323	303…373	303…423	303…473
Matrix	6.3	6.8	9.9	10.9
0.10	2.9	3.1	4.0	8.9
0.25	3.2	2.7	3.8	6.8
0.50	3.1	2.9	4.1	7.7
1.00	2.2	2.5	3.6	6.1
1.50	3.0	3.0	3.8	9.7
2.00	2.6	2.6	3.6	7.5

a)

b)

Fig. 3 – Dilatometric curves of CM for different content of the modifier $C_7H_7NO_2$: a) from 0.10 to 0.50 wt.%; b) from 1.00 to 2.00 wt.%

The experimentally obtained data of TGA and DTA analysis (Table 5, Fig. 4) demonstrate, that the beginning of the process of modified CM's destruction specimens occurs in the temperature range $\Delta T = 604\ldots616$ K, and this slightly exceeds the initial temperature of mass loss for the original epoxy matrix.

Table 5 – Heat resistance of CMs modified with 4-aminobenzoic acid ($C_7H_7NO_2$)

The content of modifier $C_7H_7NO_2$, q, wt.%	T_0, K	T_5, K	T_{10}, K	T_{20}, K	T_f, K	ε_m, %
Matrix	600	619	626	645	734	73.3
0.10	604	622	630	653	745	74.0
0.25	604	625	634	645	796	77.0
0.50	604	619	629	652	774	73.7
1.00	615	627	635	644	791	77.3
1.50	608	622	631	642	778	73.0
2.00	615	627	636	649	807	76.0

Note: T_0 – the initial temperature of mass loss (beginning of destruction); T_5, T_{10}, T_{20} – mass loss temperatures (5 %, 10 %, 20 %); T_f – the final temperature of mass loss (completion of destruction); ε_m – the relative mass loss

The values of the temperatures at which mass loss occurs (5...20 %) are also higher than existing in the modified polymer matrix. It should be noted, that the process of mass loss of CM with different content of $C_7H_7NO_2$ modifier was observed in the temperature range $T = 619\ldots653$ K. At the same time, the temperature range for the matrix is $T = 619\ldots645$ K. Completion of the process of destruction of the original polymer – $T_f = 734$ K, which is smaller than the value of the final temperature of mass loss of CM with different volume of 4-aminobenzoic acid ($T = 746\ldots808$ K). At the same time, the indicators of relative mass loss are in the range $\varepsilon_m = 73.0\ldots77.3$ %. The given analysis shows the relative temperature stability of CM's, which are modified with 4-aminobenzoic acid.

In addition, exothermic effect is important thermophysical characteristic of polymer materials. The analysis of DTA curves shows that the onset and termination of exothermic effects were observed in the temperature range $\Delta T = 593\ldots683$ K (Table 6). At the same time, the minimum value of the initial temperature of the exoeffect is set for CM with a modifier content $q = 0.50$ wt.%.
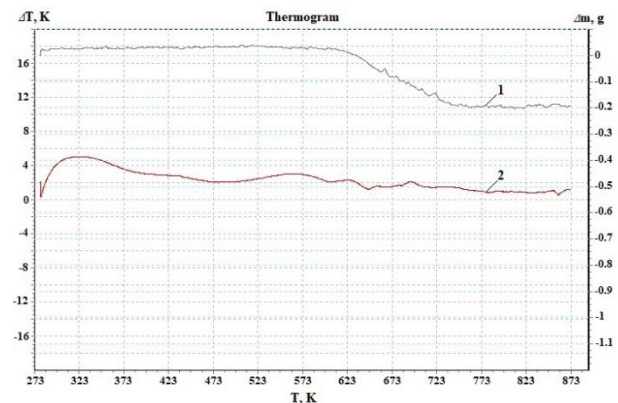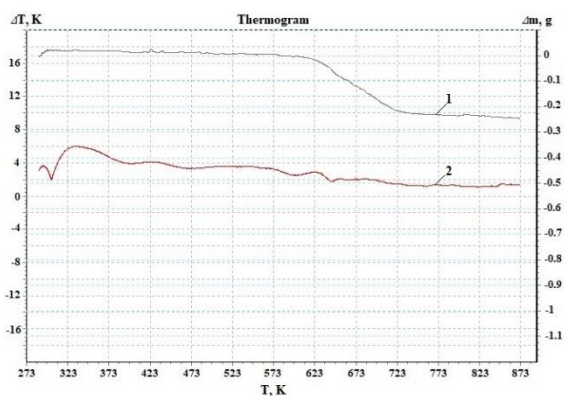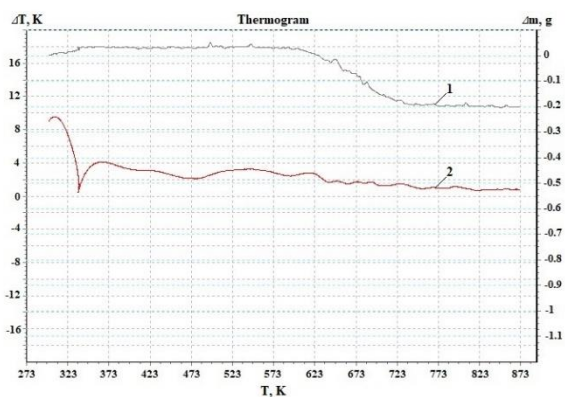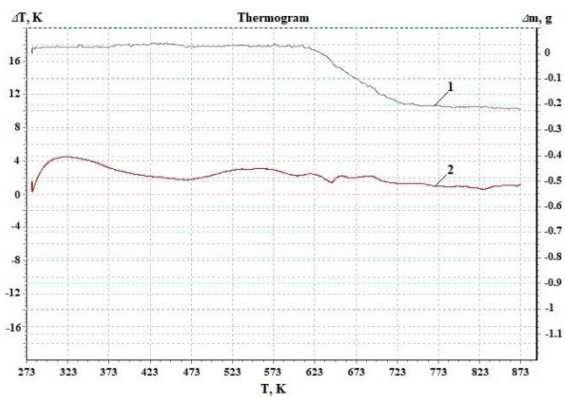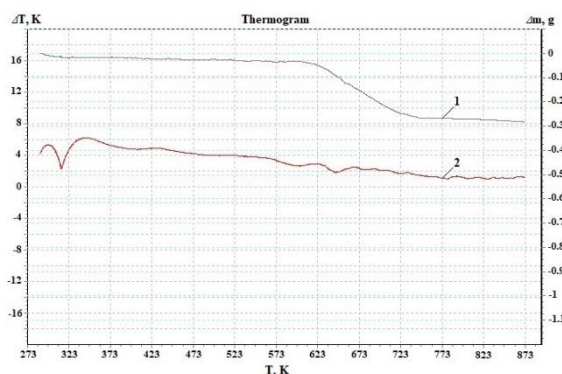

a)
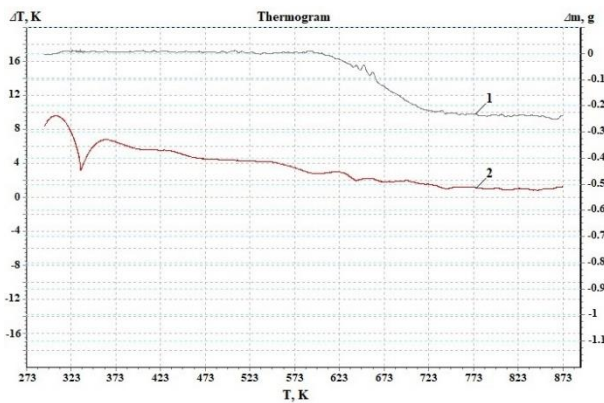
b)

c)

d)

e)

f)

g)

Fig. 4 – Thermogravimetric (1) and differential-thermal (2) analysis polymer matrix and CM with modifier content C_7H_7NO2, q, wt.%: a) unmodified matrix (control specimen); b) 0.10; c) 0.25; d) 0.50; e) 1.00; f) 1.50; g) 2.00

Table 5 – Value of temperature intervals of exothermic effects of CM

The content of modifier $C_7H_7NO_2$, q, wt.%	Temperature intervals of exoeffects				Maximum value of exoeffect, T_{max}, K
	T_i, K	T_f, K	ΔT_1, K	ΔT_2, K	
Matrix	598	683	85	2	618
0.10	598	641	43	1	621
0.25	601	644	42	1	623
0.50	593	639	46	1	617
1.00	601	643	41	1	616
1.50	597	642	45	1	623
2.00	601	644	43	1	621

Note: T_i – the initial temperature of the exoeffect; T_f – the final temperature of the exoeffect; ΔT_1 – temperature interval of the exoeffect; ΔT_2 – temperature difference between the specimen, in which transformations occur, and a standard in which there are no transformations.

In turn, the maximum value of the final temperature of the exoeffect was observed for the unmodified polymer matrix. It should be highlighted, that practically stable temperature range of the exoeffect of the modified specimens ($\Delta T_1 = 42…47$ K), which is almost twice less in relation to the temperature interval of the matrix ($\Delta T_1 = 86$ K). The maximum value of the exoeffects of the studied specimens is $T_{max} = 616…623$ K. At the same time, the deviation of the values from the maximum of the exoeffect of the epoxy matrix $T_{max} = 618$ K is $\Delta T_{max} = 1…5$ K. The analysis of the temperature intervals

of the exoeffects of the original epoxy matrix and the modified specimens gives grounds for arguing that the CM with the modifier's content $q = 0.25$ wt.% and $q = 2.00$ wt.% is characterized by the best thermal physical properties.

4. CONCLUSIONS

The results of experimental researches indicates the effectiveness of usage of the modifier 4-aminobenzoic acid ($C_7H_7NO_2$) in epoxy compositions. As a result of the work, a modified epoxy material with improved thermal physical properties was developed for an increasing of the service life of river and sea transport. The following can be stated.

Analysis of the effect of the modifier on the heat resistance of polymer composites shows a significant increase in its indicators. In particular, for the content of 4-aminobenzoic acid in the amount of 0.10 wt.%, the heat resistance of the composites is increasing in comparison with the unmodified epoxy matrix, from 341 K to 360 K. A small content of the modifier

$q = 0.01\dots1.00$ wt.% leads to insignificant changes in heat resistance and glass transition temperature of formed CM. With a further increasing of the modifiers' concentration to $q = 1.50$ wt.% and $q = 2.00$ wt.%, the decreasing of CM properties' parameters was observed. The research's results of the thermal coefficient of linear expansion gives opportunity to state that the TCLE indicators of the composites with different modifiers' content do not exceed the TCLE values in different temperatures' test ranges of the original matrix.

As we consider the broader implications and future directions, the integration of nanomaterials, could offer an avenue for optimizing the modifier's impact. The unique properties of nanomaterials at the nanoscale may contribute to fine-tuning the thermal characteristics and overcoming potential diminishing returns observed at higher concentrations. This perspective invites future research endeavors to explore the synergies between traditional modifiers and nanomaterials, aiming for a balanced enhancement in both structural and thermal performance.

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Підвищення ресурсу водного транспорту під впливом теплового поля шляхом використання модифікованих епоксидних наноматеріалів

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У роботі представлені результати експериментального дослідження впливу природи нанонаповню- вача на теплофізичні властивості композиційних матеріалів. Полімерні композиційні матеріали на ос- нові епоксидіанового олігомеру ЕД-20 модифікували 4-амінобензойною кислотою. Досліджено темпе- ратуру текучості Мартенса та визначено температуру склування розроблених матеріалів. Для встанов- лення впливу теплового поля на структурні процеси композиційних матеріалів досліджено тепловий коефіцієнт лінійного розширення матеріалів. У зв'язку з необхідністю вивчення впливу температури на фізико-хімічні процеси та перетворення в полімерних композитах використано сучасні методи тер-

мічного аналізу – диференціально-термічний та термогравіметричний аналіз. На основі аналізу експериментальних даних визначено оптимальний вміст 4-амінобензойної кислоти q = 0,10 мас.% на 100 мас.% олігомеру ЕД-20, що призводить до покращення теплофізичних властивостей епоксидних композиційних матеріалів.

Ключові слова: Епоксидна композит, Наповнювач, Нанонаповнювач, Нанорозмірні структури, Термостійкість, Термічний коефіцієнт лінійного розширення, Термічний опір.